Mathematical Modeling of Odor Deterioration of Millet (*Pennisetum glaucum*) Dough (*fura*) as Affected by Time-Temperature and Product Packaging Parameters

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ABSTRACT

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Consumer panelists evaluated the influence of temperature (5, 25, 35°C), storage time, and packaging material (aluminum foil or low-density polyethylene [LDPE]) on the sensory quality of millet dough (*fura*). The quality attributes of interest were mold growth, color, odor, and moisture. The odor sensory attribute changed significantly ($P < 0.05$) during storage and was influenced by storage time and temperature. Interactive effect of packaging-temperature and temperature-time affected odor changes significantly. A kinetic reaction model was used to analyze odor sensory data. The degradation of pleasant odor followed a first-order reaction, and the temperature dependence of these reactions indicated an Arrhenius relationship. While an activation energy (E_A) of 29.98 kJ/mol was calculated for samples stored in aluminum foil, a higher value of 49.04 kJ/mol was obtained for samples stored in LDPE. The negative values of enthalpy (ΔH^*) in both packages indicate that deterioration change in *fura* is exothermic. The higher values of E_A and free energy of activation (ΔG^*) for *fura* stored in LDPE packages indicate a large change in quality. However, the lower values of free energy of activation (ΔG^*) and negative values of entropy of activation (ΔS^*) for *fura* stored in aluminum foil packages indicate a product that is more thermally stable, and odor deterioration is less influenced by temperature. There is no significant difference ($P > 0.05$) in the shelf life of *fura* stored at 20, 25, and 35°C in the two packaging materials. However, there were significant differences in shelf life of *fura* stored at 5°C.

Fura is a thick porridge or semisolid dumpling cereal meal that is a traditional staple food in West Africa. It is produced mainly from pearl millet (*Pennisetum glaucum*) flour, blended with spices, compressed into flour balls, and boiled for 30 min. While still hot, the cooked balls are pounded to a smooth, slightly elastic, cohesive dough. The dough is reshaped into small balls using flour on a calabash gourd and rolling by hand to give the final product. The cooked dough balls are broken up and made into porridge by mixing with fermented whole milk (*kindrimo*) or fermented skimmed milk (*nono*) (Jideani and Wedzicha 1994). The most common method of packaging and marketing *fura* is by selling the *fura* balls (25–30 g) in a Chinaware dish with a cover; as such, it is sold without adequate packaging material. The processors and retailers of *fura* are primarily concerned with having a container for their food that provides protection during a generally short shelf life during local distribution (Jideani et al 2001). As distribution routes for *fura* become longer, there is a need for increased shelf life and better packaging of *fura*.

Appearance is the most important attribute influencing the consumer to accept a food product (IFT 1990; Vankerschaver et al 1996a). Purchase qualities include appearance (color, size, shape, absence of defects), firmness-to-the-touch, and aroma (Vankerschaver et al 1996b). Consumption qualities include flavor (resulting from taste and aroma) and texture perceived as mouthfeel (Shewfelt 1993). For products like *fura*, appearance is the first and most important attribute evaluated by the consumer. Microbial activity and chemical reactions cause sensory decline with off-flavors, discoloration, and loss of firmness. Water loss not only results in direct weight loss, but also causes undesired changes in appearance (cracking) and texture (hardening).

Food systems are highly complex, and precise time-temperature related quality change mechanisms are not fully understood. Hence, all time-temperature induced changes cannot be included within one mathematical model. Generally, only a single index of quality is considered in the modeling procedure. The index of quality may be any physical, chemical, enzymatic, sensory, or microbial characteristic that is detectable and predominant within the food and has importance in relation to product definition and acceptance (Wells et al 1987). Because foods are destined for human consumption, the index of quality is most appropriately defined by sensory perception. *Fura* has limited storage life, lacks adequate packaging, and has undocumented sensory quality standards or acceptability limits. Our objective was to quantify and model the sensory quality with the strongest relationship to storage treatment. The effects of temperature and packaging materials on sensory quality changes were determined.

MATERIALS AND METHODS

Materials

Fresh *fura* samples were purchased from a local market in Bauchi, Nigeria, and prepared according to the traditional method (Fig. 1). The effect of temperature, storage time, and two types of packaging materials (aluminum foil, 0.02 mm thick, and low-density polyethylene [LDPE], 0.01 mm thick) were evaluated. Water vapor transmission rate for LDPE is 50–200 g/mil/m^2/24 hr at 20°C; water vapor transmission rate for aluminum foil is practically zero (Gilbert 1985). The permeability of LDPE film to oxygen is 3,900–13,000 cm^3/m^2/mil/day at 1 atm (Zagory and Kader 1988). *Fura* samples (5 g) in groups of 10 were sealed in aluminum foil or LDPE packages and stored at 5, 25, and 35°C for three days. The temperatures were controlled using an incubator set at 25 or 35°C. Samples were evaluated for sensory attributes by consumer panelists in duplicate at 12-hr intervals over 72 hr.

Sensory Evaluation

Sensory quality attributes of the stored samples were evaluated by a 10-member consumer panel using modified quantitative descriptive analysis (QDA) (Vankerschaver et al 1996a). The preliminary stage in QDA is the collection of descriptors for appearance and the development of an appropriate evaluation sheet (Zook and Pearce 1988). However, in our study, the important sensory quality attributes were identified in advance, which shortened training to a session on the proper use of the score sheet. The quality attributes of interest and the sequence to evaluate the attributes were mold growth, color, odor, and moisture. The panel included students and staff

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from the University. Table I details the age range and gender distribution of the panelists. All panelists were *fura* consumers. Panelists received the samples on white plastic plates after the packaging material had been removed. Samples were judged in an isolated room under diffused sunlight at room temperature (Biradar et al 1985) over a period of three days. The judges indicated their perception of each quality attribute using 15-cm unstructured lines. Descriptors on both ends of the line scale were (on the left) terms were associated with a low intensity score such as not moldy, light, pleasant odor, and dry; and (on the right) terms associated with a high intensity score such as very moldy, dark, strong off-odor, and soggy. Panel members scored the perceived intensity of each attribute by placing a vertical line across the unstructured line. Panel scores were measured in centimeters from the left end of the line scale to the mark of the panelist.

Data Analysis

Homogeneous variances or homoscedasticity is a necessary prerequisite for (linear) regression models. Therefore, a reduction in variability within the sensory panel was obtained by a normalization method described by Davis and Goldsmith (1988). Panelist scores were weighted by the inverse of the associated variance. The normalized scores were evaluated with analysis of variance (ANOVA) (SPSS Inc., Chicago, IL) to determine the quality attribute that had the strongest relation to storage time, temperature, and packaging material. This quality attribute was then analyzed with a kinetic reaction model. To facilitate the kinetic model, a value of $(15 - R_{\text{norm}})$, where R_{norm} is the normalized mean score, was used for further analysis. Thus, the quality attribute decreased from an initial maximum score. This procedure was consistent with the methods described by Vankerschaver et al (1996a) and Wells and Singh (1988).

Mathematical Modeling of Quality Deterioration

For the quality deterioration of *fura*, first-order kinetics was assumed; that is, for the quality factor Q, the rate of change during storage is given by the equation

$$dQ/dt = -kQ \tag{1}$$

where dQ/dt = rate of change of Q/unit time (t), k = rate constant, Q = value of Q at time t. Integrating Equation 1 gives $\ln Q = \ln Q_0 - kt$. When the values of $\ln Q$ are plotted against time, the reaction rate constant k can be obtained from the slope. The influence of a constant temperature on the reaction rate constant can be described using the Arrhenius equation (Vankerschaver et al 1996a)

$$\ln\left(k_{\text{q}}\right) = \ln\left(k_{\text{q}ref}\right) + \left[{E_{\text{a}}}\big/{R} \cdot \left(\frac{1}{T_{ref}} - \frac{1}{T}\right)\right] \tag{2}$$

where k_{q} = reaction rate constant; k_{qref} = quality rate constant at reference temperature (T_{ref}); T = absolute temperature (in degrees K); E_{a} = apparent activation energy (in J/mol); R = universal gas constant (8.314 J/mol K). The reference temperature (293 K or 20°C) was the mean value of the temperatures studied (Van Boekel 1996).

RESULTS AND DISCUSSION

Effect of Packaging Material, Time, and Temperature on Sensory Quality of *Fura*

Table I shows the demographic data for the consumer panelists. Most of the panelists (90%) were between the ages of 18 and 45, 70% were males. The high number of panelists with undergraduate experience was not unusual for a university community. The effect of packaging material, storage temperature, storage time, and their interaction on the sensory quality of *fura* is detailed in Table II. The packaging materials and temperature had significant effect ($P < 0.05$) on the color of the samples.

Storage time had a significant effect ($P < 0.05$) on mold growth but no significant interactions were observed for mold growth (Table II). Packaging had no significant effect ($P > 0.05$) on the changes in odor and moisture. However, significant effects were observed for storage temperature and time. There was a significant temperature-time interaction ($P < 0.05$) for color, odor, and moisture.

The odor sensory attribute changed significantly ($P < 0.05$) during storage and was influenced by storage time and temperature (Table II). Interactive effects for packaging-temperature and temperature-time affected odor changes significantly. An earlier study of *fura* production in some northern states of Nigeria identified two major signs of spoilage of *fura* as off-odor and mold growth (Jideani et al 2001). Mold growth would not be an effective index of quality because some consumers wash off the mold and slime on the surface of the *fura* balls and consume the product as they would normally. Therefore, changes in odor (pleasant to strong off-odor) were selected as the index of quality in the modeling procedure.

TABLE I
Demographic Data for Consumer Panelists (n = 10)

Variable	Percentage
Sex	
Male	70
Female	30
Age	
18-25	40
26-35	20
36-45	30
46-55	10
>55	0
Education	
University graduate	30
Undergraduate	50
Postgraduate	10
College graduate	10

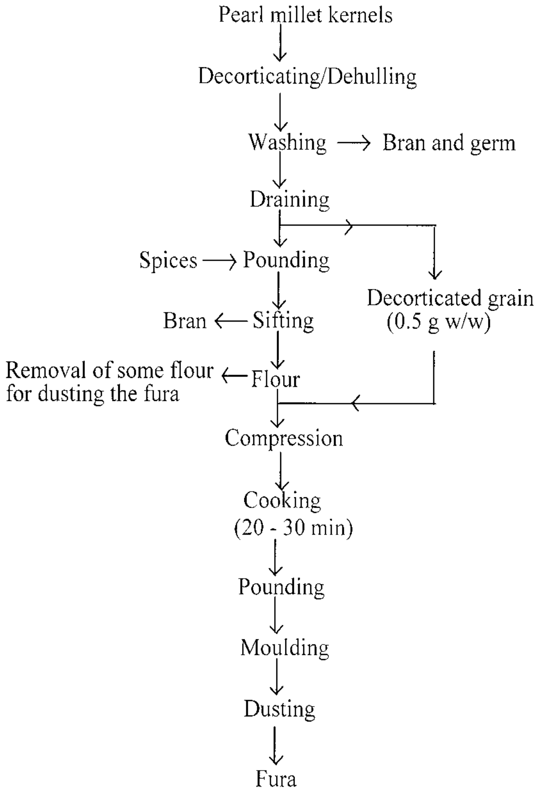

Fig. 1. Traditional *fura* production process.

TABLE II
Analysis of Variance for Packaging Material, Storage Temperature, Storage Time, and Their Interactions on Sensory Quality Attributes of *Fura*

Sensory Attributes and Factors	df	Sum of Squares	Mean Square Value	F-Value[a]
Mold				
Packaging material	1	77.09	77.09	2.51
Temperature (°C)	2	8.66	4.33	0.14
Time	6	586.67	98.28	3.20*
Package × Temp.	2	10.80	5.40	0.18
Package × Time	6	68.65	11.44	0.37
Temp. × Time	12	299.60	24.97	0.81
Color				
Packaging material	1	28.34	28.34	5.64*
Temperature (°C)	2	99.73	49.87	9.92*
Time	6	68.17	11.36	2.26
Package × Temp.	2	35.23	17.62	3.50*
Package × Time	6	21.15	3.52	0.70
Temp. × Time	12	210.10	17.51	3.48*
Odor				
Packaging material	1	2.10	2.10	1.42
Temperature (°C)	2	35.62	17.81	12.00*
Time	6	430.88	71.81	48.40*
Package × Temp.	2	25.99	13.0	8.76*
Package × Time	6	6.81	1.13	0.77
Temp. × Time	12	77.15	6.43	4.33*
Moisture				
Packaging material	1	3.09	3.09	0.83
Temperature (°C)	2	41.03	20.52	5.47*
Time	6	262.80	43.80	11.69*
Package × Temp.	2	3.86	1.93	0.52
Package × Time	6	34.38	5.73	1.53
Temp. × Time	12	141.50	11.79	3.15*

[a] * = Significant effect at $P < 0.05$.

TABLE III
Kinetics of Odor Development in *Fura* During Storage[a]

Packaging	Temp. (°C)	ln Q_0	k (hr^{-1})	r^2
Aluminum foil	5	2.62	0.007	0.89
	25	2.97	0.019	0.76
	35	2.97	0.023	0.83
LDPE	5	2.62	0.004	0.82
	25	2.71	0.019	0.99
	35	3.09	0.031	0.77

[a] Q_0 = odor value at zero storage time; k = rate constant (hr^{-1}); r^2 = coefficient of determination. LDPE = low-density polyethylene.

Mean normalized responses ($15 - R_{\text{norm}}$) where R_{norm} is the normalized mean score of odor of *fura* stored in aluminum foil or low-density polyethylene (LDPE) packages at 5, 25, and 35°C were compared (Fig. 2). After 12 hr of storage, there was a significant drop in quality in all the samples except the sample stored at 5°C in LDPE.

Kinetics of Quality Deterioration

The first-order model (Equation 1) was used to describe the sensory quality deterioration of *fura* with respect to disappearance of pleasant odor. Quality deterioration rate constant (slope) and intercept were estimated by linear regression using the mean normalized panel responses (Table III). The coefficient of determination (r^2) > 0.70 shows that decrease in pleasant odor followed first-order reaction kinetics during storage. For both packaging materials, the rate constant increased as storage temperature increased. The low values of the rate constants obtained for samples stored at refrigeration temperature indicate that the odor of *fura* is relatively stable under refrigeration.

Effect of Temperature on the Rate Constant

The rate constants were plotted against the reciprocal of the absolute temperature according to the Arrhenius equation. Figure 3 shows the results for the two packaging materials. It is evident that the relationship (ln k vs. $1/T$) is linear in the given temperature range, which makes it possible to determine the activation energy (E_A). Table IV gives the E_A (obtained by regression), the quality rate constant at reference temperature (k_{qref}), and the coefficient of determination (r^2) of the straight lines. The high values of the latter demonstrated that the experimental results could be described by the Arrhenius equation over the temperature range studied. While an E_A of 29.98 kJ/mol was calculated for samples stored in aluminum foil, a higher value of 49.04 kJ/mol was obtained for samples stored in LDPE. According to Fu et al (1991), the E_A values for growth of microbes are 60–120 kJ/mol. The values obtained in this study suggest that changes in odor were not necessarily due to microbial growth. The characteristic mousy, acidic odor in ground pearl millet during brief storage was associated with oxidative rancidity (Reddy et al 1986) as well as hydrolytic changes in lipids (Lai and Varriano-Marston 1980). There was no significant difference ($P < 0.05$) in the rate constant at reference temperature (k_{qref}) for the two packaging materials: aluminum foil 0.014/hr and LDPE 0.012/hr.

Thermodynamic Considerations

The E_A value allowed determination of different thermodynamic parameters such as the enthalpy (ΔH^*), the entropy (ΔS^*), and the free energy (ΔG^*) of activation according to the expressions (Sánchez et al 1992):

$$\Delta H^* = E_A - RT \tag{3}$$

$$\Delta S^* = R\left(\ln A - \ln \frac{k_B}{h_p} - \ln T\right) \tag{4}$$

$$\Delta G^* = \Delta H^* - T\Delta S^* \tag{5}$$

where R = universal gas constant, ln A = the ordinate intersection when regression analysis is applied to the plot obtained in calculation of E_A, k_B = Boltzmann constant (1.38×10^{-23} JK^{-1}), h_p = Planck constant (6.626×10^{-34} Js) and T = absolute temperature.

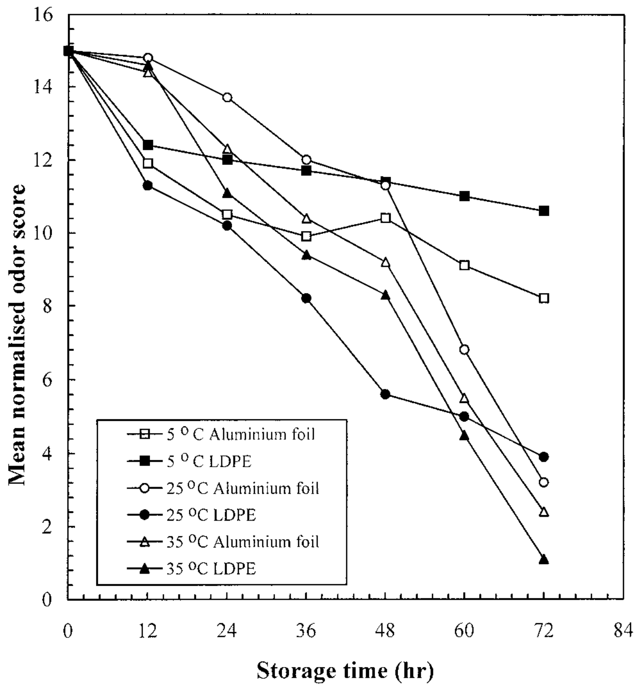

Fig. 2. Changes in normalized mean score (15 – R_{norm}) for odor of *fura* stored in aluminum foil or low-density polyethylene (LDPE) packages at 5, 25, and 35°C.

The result of the thermodynamic parameters for *fura* in aluminum foil and LDPE packages are shown in Table V. The negative values of enthalpy (ΔH^*) in both packages indicate that deterioration changes in *fura* are exothermic. The metabolic processes going on in *fura* will use up the oxygen in the package at the same time that CO_2 is being produced by carbohydrate, protein, and fat metabolism (Labuza 1996). Because the metabolism of fat and carbohydrate produces water, and wet food has a high vapor pressure, the humidity in the food package will increase and allow growth of molds and yeasts (Labuza 1996), leading to unpleasant odor. The values of the activation parameters obtained suggested that there was another process determining the rate of deterioration of the pleasant odor in *fura*. This hypothesis was particularly supported by the negative entropy of activation (ΔS^*) (Dannenberg and Kessler 1988) for the two packaging materials. According to the theory of the activated complex, a substance in a state of activation can only have a negative entropy of activation if degrees of freedom of translation or rotation are lost during the formation of the activated complex. The transitional state is then in a higher state of order than the reactant particles of which it is composed. The higher values of E_A and free energy of activation for *fura* stored in LDPE indicate that *fura* in LDPE packages experiences a large change in quality, and odor deterioration was more influenced by temperature. However, the lower values of free energy of activation (ΔG^*) and negative values of entropy of activation for *fura* stored in aluminum foil indicate that *fura* in aluminum foil packages were more thermally stable, and odor deterioration was less influenced by temperature.

Effect of Time-Temperature Combination on Odor of *Fura*

The estimates of k_{qref} and E_A, allowed the index of quality (Q) to be predicted for combinations of storage time (t) and temperature (T). The time-temperature relationship of *fura* odor is shown in Figs. 4 and 5. In these three-dimensional representations, the *fura* odor sensory scores predicted by the first-order reaction and the Arrhenius equation are depicted as quality response surface patterns (Wells et al 1987). These provide a geometrical representation of the behavior of pleasant odor within the experimental design.

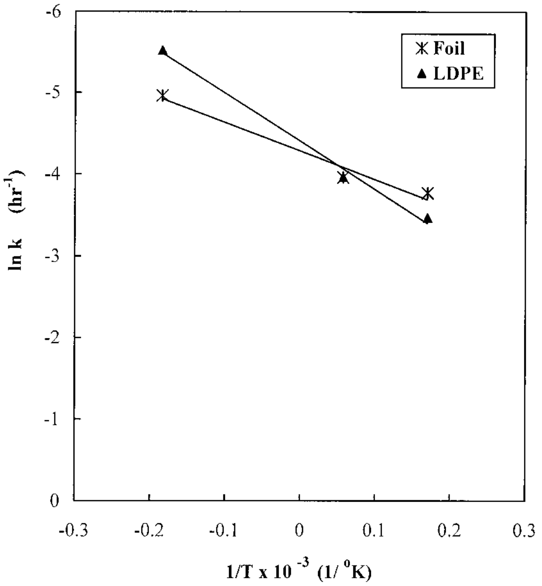

Fig. 3. Arrhenius plot of first-order reaction rate constants for pleasant odor changes in *fura* during storage in aluminum foil or low-density polyethylene (LDPE) packages.

TABLE IV
Parameters of Arrhenius Equation for Odor Changes in *Fura* During Storage[a]

Packaging	Temp. (°C)	E_A (kJ/mol)	k_{qref}	r^2
Aluminum foil	5–35	29.98	0.014	0.988
LDPE	5–35	49.04	0.012	0.997

[a] LDPE = low-density polyethylene. Reference temperature (T_{ref}) set at 293 K (20°C); E_A = activation energy; k_{qref} = quality rate constant at reference temperature (T_{ref}); r^2 = coefficient of determination.

The pleasant odor decreased with increase of temperature and storage time in both packaging materials. The temperature-time interaction resulted in quadratic decreases of odor deterioration. The effect of temperature on the *fura* in LDPE is more intense (shown by the steepness of the temperature curve) than that in aluminum foil (Fig. 5). The shelf life of a product is defined as the period during which the sensory quality remains suitable for consumption or the process intended (Wells and Singh 1988). This implies that there is some limiting threshold of quality beyond which a product is unsuitable for consumption. From Figs. 4 and 5, the limiting odor score after which there is more rapid quality change is 12. Recall that to facilitate the kinetic model, a value of (15 – R_{norm}), where R_{norm} is the normalized mean score, was taken for further analysis. Therefore, the normalized mean score corresponding to the limiting threshold of 12 is 3. From Figs. 4 and 5, the *fura* in aluminum foil attained off-odor of ≥12 after 69, 36, 30, and 15 hr at 5, 20, 25, and 35°C, respectively. *Fura* stored in LDPE attained off-odor score of ≥12 after 60, 39, 30, and 15 hr at 5, 20, 25, and 35°C, respectively. There is no significant difference ($P > 0.05$) in the shelf life of *fura* in the two packaging materials at 20, 25, and 35°C. However, significant differences existed in shelf life at 5°C.

CONCLUSIONS

The packaging materials, storage time, and temperature had significant effect ($P < 0.05$) on the color and odor of *fura*. Using changes in odor as the index of quality, there was a significant drop in quality in all samples after 12 hr of storage, except for the sample stored at 5°C in LDPE. Kinetic studies showed that odor of *fura* is relatively stable under refrigeration (5°C). The negative values of enthalpy obtained from aluminum foil and LDPE indicate

TABLE V
Thermodynamic Parameters for Thermal Stability of *Fura* During Storage[a]

Packaging	Temp. (°C)	E_A (kJ/mol)	ΔH^* (cal/mol)	ΔS^* (cal/K mol)	ΔG^* (kcal/mol)
Aluminum foil	5	29.98	–2281.31	–279.95	75.55
	25	29.98	–2447.59	–280.52	81.15
	35	29.98	–2530.73	–280.80	83.96
LDPE	5	49.04	–2262.25	–280.92	75.83
	25	49.04	–2428.53	–281.50	81.46
	35	49.04	–2511.67	–281.77	84.27

[a] E_A = activation energy; ΔH^* = enthalpy change; ΔS^* = entropy change; ΔG^* = free energy change. LDPE = low-density polyethylene.

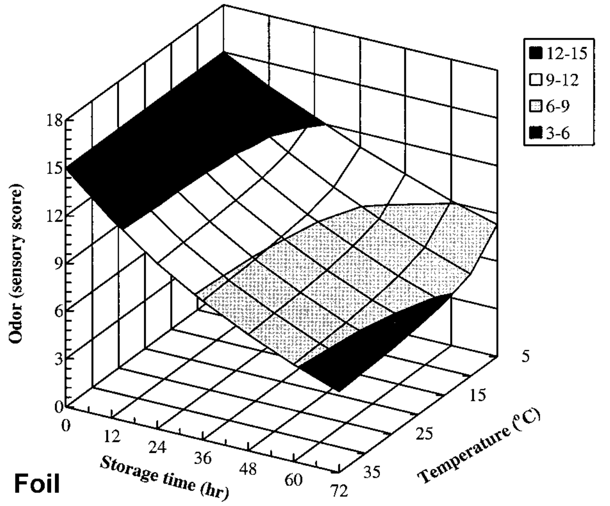

Fig. 4. Response surface pattern for *fura* odor (pleasant) stored in aluminum foil packages as predicted by first-order kinetics and Arrhenius equation.

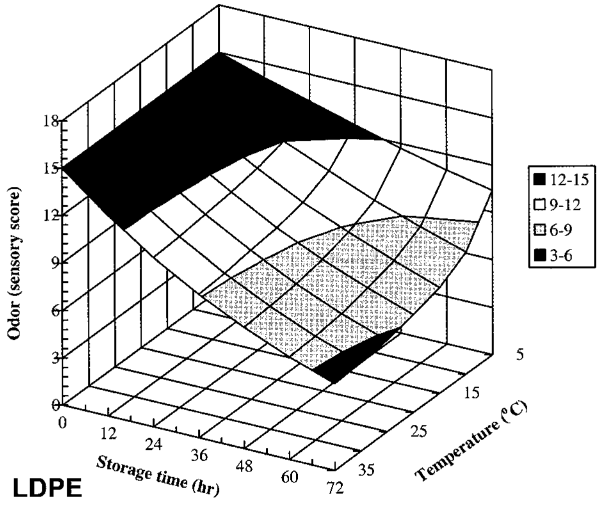

Fig. 5. Response surface pattern for *fura* odor (pleasant) stored in low-density polyethylene (LDPE) packages as predicted by first order kinetics and Arrhenius equation.

that deterioration changes in *fura* are exothermic. The lower values of free energy of activation and entropy of activation for *fura* stored in aluminum foil indicate that *fura* in this packaging were more thermally stable and odor deterioration was less influenced by temperature. Although a higher E_A (49.04 kJ/mol) was obtained for LDPE than for aluminum foil, this value is lower than the range 60–120 kJ/mol required for microbial growth. Time-temperature effect showed a decrease in pleasant odor with increase in temperature and storage time in both packaging materials. However, significant differences existed in shelf life at 5°C. It is, therefore, expected from this study that *fura* in aluminum foil or LDPE would store well at temperatures of 5–20°C.

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